

AMENDED SPECIFICATION

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Reprinted as amended in accordance with the Decision of the Patents Appeal Tribunal acting for the Comptroller-General, dated the tenth day of February 1956, under Section 14, of the Patents Act, 1949.

PATENT SPECIFICATION

661,685



Date of Application and filing Complete Specification: Feb. 10, 1949.

Nos. 3720/49 and 3721/49.

Application made in Switzerland on Feb. 20, 1948.

Application made in Switzerland on Dec. 21, 1948.

Complete Specification Published: Nov. 28, 1951.

Index at acceptance:—Class 1(ii), A13; and 1(iii), N4a(1a:x), N(34:40:x1b).

COMPLETE SPECIFICATION

ERRATUM

AMENDED SPECIFICATION NO. 661,685

POOR
QUALITY

Page 6, line 130, for "hydrogen - containing" read "hydrocarbon - containing".

THE PATENT OFFICE,
29th November, 1957

DB 00964/2(2)/3611 160 11/57 R

ERRATUM

SPECIFICATION NO. 661,685

The heading under AMENDED SPECIFICATION should read "Reprinted as amended in accordance with the Decision of the Patents Appeal Tribunal, dated the tenth day of February, 1956 under Section 14, of the Patents Act, 1949."

THE PATENT OFFICE,
20th December, 1956

DB 41460/1/3638 100 12/56 R

30 It is known that these volatile chlorides can be transformed with oxygen, air or other oxygen-containing gases at temperatures above 500°C to the respective oxides. In order to obtain products which are completely oxidized, i.e. which do not contain any non-decomposed chlorides nor oxychlorides, it is absolutely necessary that the oxide particles, 35 more particularly in statu nascendi, pass through a temperature zone of at least 800, but preferably 950 to 1100°C. The reaction of the volatile chlorides with oxygen in general is exothermic, the heat is not sufficient 40 actually to reach the required high temperatures, starting from the cold or only moderately preheated reaction components in the

70 it has been attempted, therefore, to feed the required additional heat to the decomposition process entirely or partly by separately preheating the gases to be reacted, i.e. chloride on the one hand and oxygen or oxygen-containing gases on the other hand, to temperatures of 800 to 1000°C and then exposing 75 them to the reaction.

80 However, in carrying out this process on a technical scale, the very preheating causes considerable difficulties. Both the chlorides and the oxygen-containing gases are extremely aggressive at these temperatures and attack the known metals and alloys to such an extent that only ceramic materials can be used as a material for constructing the preheaters, feed

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COMPLETE SPECIFICATION

Improvements in or relating to the Production of Finely Divided Oxides of Titanium, Silicon, Zirconium and Aluminium

We, SÄUREFABRIK SCHWEIZERHALL, of Schweizerhalle, Kanton Baselland, Switzerland, a body corporate organized according to the Laws of Switzerland, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to certain improvements in or relating to the production of finely divided oxides of Ti, Si, Zr and Al by the decomposition of the corresponding volatile chlorides by means of oxygen-containing gases at high temperatures with formation of flames.

The term "oxygen-containing gases" as used in this specification and claims includes pure oxygen or air or other gases containing free oxygen, but is not intended to include gases or vapours of substances such as carbon dioxide or alcohol containing only combined oxygen.

"Volatile chlorides" of Ti, Si, Zr and Al in the sense of this specification are the chlorides which are sublimable and distillable and can be volatilized at temperatures below 500°C.

It is known that these volatile chlorides can be transformed with oxygen, air or other oxygen-containing gases at temperatures above 500°C to the respective oxides. In order to obtain products which are completely oxidized, i.e. which do not contain any non-decomposed chlorides nor oxychlorides, it is absolutely necessary that the oxide particles, more particularly in statu nascendi, pass through a temperature zone of at least 800, but preferably 950 to 1100°C. The reaction of the volatile chlorides with oxygen in general is exothermic, the heat is not sufficient actually to reach the required high temperatures, starting from the cold or only moderately preheated reaction components in the

reaction zone; in fact, it is not even sufficient to maintain said temperature. More particularly, the very intensive radiation of the oxide particles formed produces a very considerable loss of heat within the reaction zone. Hence, it follows that it is necessary to feed additional heat.

It is particularly difficult to produce oxides of highest fineness which meet the requirements of the pigment color industry which for most of the oxides to be manufactured from the volatile chlorides represents the main field of application. The simplest way of feeding the additional heat would consist in heating the reaction chamber from outside, a measure which would be more suitable in the flameless decomposition of the chlorides in the form of a diffuse reaction. But in the hitherto known processes with separate feeding of the reaction components, even in case of decomposition of the chlorides under formation of flames, the application of this heating method was not a success, owing to the unavoidable wall reactions with incrustation of the walls and agglomeration of the oxide particles obtained in loose condition.

It has been attempted, therefore, to feed the required additional heat to the decomposition process entirely or partly by separately preheating the gases to be reacted, i.e. chloride on the one hand and oxygen or oxygen-containing gases on the other hand, to temperatures of 800 to 1000°C and then exposing them to the reaction.

However, in carrying out this process on a technical scale, the very preheating causes considerable difficulties. Both the chlorides and the oxygen-containing gases are extremely aggressive at these temperatures and attack the known metals and alloys to such an extent that only ceramic materials can be used as a material for constructing the preheaters, feed

pipes, nozzles etc. Another difficulty consists in the danger of obstruction of the feed pipes, since the reaction components at the required preheating temperatures react immediately as they meet each other and after a short time give rise to obstruction of the outlets of the gas feeding channels.

Another known method described in British Specification 258,313 consists in distributing volatile halogenides of a metal or silicon together with a combustible liquid or gas in an oxidizing gas, igniting the mixture to produce a flame and separating the resulting oxide from the products of combustion. Such compounds as ferric chloride, aluminium chloride, silicon chloride, titanium chloride or mixtures of such compounds may thus be brought into a flame in a finely divided condition. For example, purified coal gas is loaded with titanium chloride vapors, distributed in the air and ignited. The heat which can be produced additionally in this process by combustion of the combustible gas ought to be sufficient to maintain the reaction once initiated between combustible gas or chloride on the one hand and oxygen-containing gas on the other hand, even in case of a moderate preheating of the initial products. It has been found, however, that a mixture consisting of a combustible gas and chloride if only slightly preheated, is very difficult to ignite in an oxygen-containing atmosphere, that the flame once formed tends to go out again and, moreover, that chloride vapors even in small quantities increase the ignition temperature of combustible gases considerably—up to several 100 degrees centigrade. The process just described therefore can be carried out only if large quantities of combustible gases in proportion to the quantity of chloride are used, whereby the process thus becomes uneconomical.

Finally, the decomposition of silicon tetrachloride was attempted in such a way that the same, mixed with hydrogen and a small quantity of air, is ignited in burners of small dimensions under rotary cylinders and decomposed, drawing the additional oxygen required for the formation of silicon dioxide from the outer atmosphere. In order to carry out this process, a large excess of hydrogen is required, and owing to the danger of explosion only small quantities of oxygen-containing gases can be admixed to the silicon chloride vapor. Moreover, it is also known that this method after a short time leads to quick obstruction of the burner in case of metal chlorides having a higher reaction speed than silicon tetrachloride with the steam forming in the burning flame. Moreover, the output of such a burner is very small.

Now, it has been found that all these difficulties can be overcome by the following procedure:

According to the invention a process of pro-

ducing finely divided oxide of titanium, silicon, zirconium or aluminium by the decomposition by burning at high temperatures of the corresponding volatile chloride in the presence of oxygen-containing gases comprises the steps of supplying into a reaction chamber a reaction gas mixture of the chloride in the vapour state and oxygen-containing gas at a temperature above the dew-point of the chloride in the reaction gas mixture but not exceeding 500°C., a combustible gas introduced separately from the reaction gas mixture, and oxygen-containing gas additional to the oxygen-containing gas in the reaction gas mixture and introduced separately from the reaction gas mixture, the additional oxygen-containing gas introduced being at least sufficient to ensure that the combustible gas would burn under flaming conditions in the absence of the reaction gas mixture and being at least sufficient (together with the oxygen-containing gas in the reaction gas mixture) to ensure complete combustion of the chloride, the gases being so introduced into the reaction chamber and in such proportions and amounts that the combustible gas and the additional oxygen-containing gas (introduced separately from the reaction gas mixture) are at least in part admixed before such admixture meets the reaction gas mixture and that the said combustible gas and the additional oxygen-containing gas serve to maintain the burning of the reaction gas mixture in a flaming state.

Advantageously the burning decomposition of the reaction gas mixture begins at a suitable distance from the discharge opening of the reaction gases flowing into the reaction chamber, i.e. a distance which is determined by the discharge conditions of the gases which should be chosen in such a way that on the one hand obstruction of the openings through which the reaction gases are discharged into the reaction chamber, and, on the other hand, a diffuse reaction are avoided.

It is possible to add to the chloride vapor oxygen-containing gas and even pure oxygen and to heat this mixture to temperatures up to 500°C, without any reaction taking place such as it is the case with many mixtures of combustible gases with oxygen. Contrary to expectation it was found that such a mixture even with an oxygen content of any high amount can be ignited by means of the exothermic auxiliary chemical reaction of burning combustible gas in a flaming condition to form a flame and brought to a quick reaction at temperatures above 1000°C without flashing back of the flame to the outlet opening, which flame has been ignited at a suitable distance from the outlet, and without consequent explosion within the feed pipe and the mixing device. This knowledge forms the basis of the above described new method.

Also, obstruction of the feed pipe, more

particularly of the outlet opening, can be avoided in this process, since the flame by a suitable mixing ratio of the reaction gas mixture, suitable temperature and exit velocities of the reaction gases passing from their feed nozzles can be held without difficulty at some distance from the outlet opening. Within the conditions coming into consideration on a technical scale, a minimum distance of about 1 mm, preferably from 3 mm upwards, will be sufficient to prevent growing over of the outlet opening. For reasons of safety, however, the minimum distance will be made somewhat larger in practical operation. However, the ignition of the flame also must not take place at too large a distance from the outlet opening, since the reaction otherwise might take a diffuse and possibly incomplete course. According to experience the maximum distance under the above mentioned conditions amounts to about 1 meter. Since in this process the chloride vapor is exposed to the reaction in a state of being mixed at least with part of the oxygen required for its transformation with formation of flames, the reaction, once it is started, proceeds much more rapidly than in case the reaction components are mixed only during the reaction. This is of a particular importance in view of the tendency of the primarily formed oxide particles of growing further. By the process according to the invention this chance, which otherwise always exists, is not offered to them. This fact ensures the formation of a fine grain in a much higher degree than in the hitherto known processes. Also the development of heat within the flame is considerably facilitated and made more uniform. Where the reaction gas does not contain in itself the amount of oxygen required for the reaction, the missing share is fed to the reaction in the form of any oxygen-containing gas mixture.

The amount of oxygen required by the chlorides for completing the reaction exceeds the stoichiometric quantity. This excess beyond the quantity of oxygen stoichiometrically required is different with the different chlorides; with titanium chloride, e.g. only a few percent by volume are required.

A particularly fine grain of the oxide is obtained where an excess of oxygen is admixed to the chloride. The grain size grows as the oxygen percentage of the reaction gas is reduced.

An amount of oxygen, sufficient to maintain flaming combustion of the combustible gas in the absence of the reaction gas mixture is supplied separately from the reaction gas mixture. This amount need not be sufficient for the complete combustion and oxidation of the combustible gas. In such case the additional part of the oxygen required for the complete combustion of the combustible gas may also be fed admixed with the reaction gas mixture. The oxygen for the combustion

supplied separately from the reaction gas mixture is advantageously at least in part fed separately from the combustible gas. Where only part of the oxygen is so fed separately from the combustible gas, the oxygen required in addition to such partial separate oxygen feed may be fed in mixture with the combustible gas.

As a combustible gas, hydrogen, carbon monoxide, lighting gas, benzine vapors, oil vapors etc. can be used. In general, the combustible gas and the oxygen-containing gas fed separately for its combustion are advantageously delivered concentrically and possibly separately around the reaction gas current. The combustible gas, once being ignited, forms a constantly burning flame which may ignite the reaction gas mixture at a certain distance from the outlet opening, owing to the heating by the combustion products. To maintain the flame of the reaction gas mixture it is necessary that the supply of the gases for the auxiliary combustion is continuous and constant, because the reaction gas mixture has to be ignited constantly by the flame of the combustible gas. If the supply of the gases for the auxiliary reaction is interrupted, the flame of the reaction gas will become extinguished and the reaction gas mixture will react in a diffuse reaction. Where carbon monoxide is used as a combustible gas, the same can be fed directly around the reaction gas current without causing a deposit at the outlet opening of the reaction gas, since the carbon monoxide reacts so slowly with the oxygen contained in the chloride vapor that it ignites with the same only at a certain distance from the outlet opening and only there inflames the reaction gas. In this case the separately fed oxygen-containing gas may be fed around the carbon monoxide. Where elemental hydrogen-containing combustible gases are fed, on the other hand, the oxygen-containing gas is advantageously fed in the intermediate layer, for the following reason: Since the hydrogen fed in the intermediate layer would react very quickly with the oxygen contained in the chloride vapor with formation of water, which in turn would cause an immediate decomposition of the chloride, the reaction gas outlet would be obstructed in a short time.

Particularly fine oxides can be obtained with the method of feeding the gases for the auxiliary reaction concentrically around the reaction gas by discharging at least one of the gases for the auxiliary reaction in a direction intersecting with the direction of the reaction gas stream. In this manner a considerably swifter mixing of the reaction gases with the hot combustion products of the auxiliary reaction is attained than in the case where the gas streams of the auxiliary reaction are directed parallel to the reaction gas stream. By this intensive mixing the reaction gas is

very quickly heated to the reaction temperature and caused to react, whereby the formation of a very fine grain is greatly promoted.

A considerable effect can already be attained if only one, especially the outermost gas stream of the auxiliary reaction is directed into the reaction gas current. A still greater effect is obtained if both the gas streams of the auxiliary reaction are directed into the reaction gas current.

Advantageously the combustible gas and the percentage of oxygen at least partly sufficient for its combustion and possibly being fed separately can be burnt with a twirling or spinning motion around the reaction gas current flowing into the reaction chamber. In this case the spinning motion of the burning gases may be regulated in such a way that it causes simultaneously the quick mixing of the combustion products with the reaction gas. It is also possible, however, to feed the reaction gas mixture itself to the reaction chamber in a spinning motion; in this case, provided that all or only two of the gas currents have been twisted, these spinning motions may be equally or oppositely directed, in any combination.

Twisting or spinning of the gas may be applied for carrying out the decomposition by means of an auxiliary reaction both in case of parallel feed of the gas streams and in case the gas streams of the auxiliary reaction are directed into the reaction gas current.

Where the heat delivered within the reaction chamber by the auxiliary exothermic chemical reaction serves substantially only for igniting the reaction gas, the amount of heat missing for carrying out the reaction process may be delivered by a further additional source of heat within the reaction chamber or entirely or partly by the walls of the reaction chamber which are heated by special means. This is particularly advantageous where the process is carried out on a smaller scale, since in this case owing to the high loss of heat to the outside it would be necessary to produce too much heat within the reaction chamber. When carrying out the process on a larger technical scale, the thermal insulation of the reaction chamber may be made so perfect that the amount of heat to be fed additionally can be reduced to a very low amount.

The chloride vapor may be mixed with the oxygen-containing gas in various manners. The oxygen-containing gas can be fed to the still or to the sublimation chamber for the chloride to be volatilized and then the mixture can be preheated together, if necessary, in a preheater by means of high pressure steam, gas or electrically or by any other manner as known per se, but it is also possible at first to volatilize and possibly preheat the chlorides alone and only then mix them with the cold or also preheated oxygen-containing gas. It is also contemplated to superheat liquid chloride under pressure, discharge it through

a nozzle and atomize it with the oxygen-containing gas, advantageously choosing the temperature and pressure of the two components to be mixed in such a manner that the chloride component is in a gaseous state in the mixture. This mixing can be effected only shortly before entering the reaction chamber. Since all these preparatory operations, i.e. mixing, distilling, preheating, take place at temperatures below 500°C, it is possible to carry them out in metal apparatus.

These advantages and the facilitated production of fine-grained products attained by the preceding mixing afford a considerable simplification as compared to all existing methods and devices for the production of the oxides from chlorides.

Some embodiments of apparatus which may be used for carrying out the invention will be hereinafter described by way of example and purely schematically with reference to the accompanying drawings, in which:

Fig. 1 is a longitudinal section of a vertical reaction chamber;

Figs. 2 and 3 are similar sections, showing modified forms of a vertical reaction chamber;

Fig. 4 is a perspective view showing the front end of a burner.

Figs. 5 and 6 are similar views, but showing modified forms;

Figs. 7 and 8 are end views of two modified forms of burners;

Figs. 9 and 10 are sectional views of two further forms of burners.

Similar reference numerals denote similar parts in the different views.

Referring now to the drawings in greater detail, it will be seen that the device for carrying out the process according to the invention in general consists of a reaction chamber formed by heat-insulated walls (a) with feed pipe (b) for the reaction gas mixture. The reaction chamber is provided with feed ducts (c) for the components of the auxiliary chemical reaction permitting the ignition of the reaction gas, and, if necessary, may be provided with a further oxygen feed pipe (d) for delivering the additionally required oxygen. It is also possible to provide a reaction chamber with several feed pipes for the reaction gas mixture, igniting the discharging reaction gas currents by one common or preferably by several auxiliary exothermic chemical reactions.

In addition the devices shown in Figs. 1 to 3 are provided with a funnel e serving to carry away the precipitated metal oxides and an exhaust opening f for drawing off the raw end gases. Since the raw end gases still contain substantial quantities of metal oxide, they are advantageously delivered to a dust-extracting plant.

Referring now to the various figures individually

Fig. 1 shows a vertical reaction chamber

with a feed pipe c_1 for the gases serving for the auxiliary chemical reaction.

Fig. 2 shows a device with lateral concentric feed pipes c_2 and c_3 , advantageously designed in the form of a burner, for the gases of the auxiliary reaction.

Fig. 3 shows a device with annular feed channels c_4 and c_5 for the gases of the auxiliary reaction, arranged concentrically around the feed pipe b for the reaction gases. For instance, duct c may serve for feeding a combustible gas, such as carbon monoxide or hydrocarbon, while the duct c_5 serves for delivering the oxygen required for burning this gas.

Fig. 4 shows in greater detail a burner or ignition device combined with the feed pipe for the reaction gas mixture, in which the feed pipe b for the reaction gas is provided with helically shaped partition walls g imparting the required spin to the reaction gases. For the rest, the reaction chamber for installation of this burner or any of the burners shown in Figs. 5 to 10 may take any of the forms shown in Figs. 1 to 3.

Fig. 5 shows a similar burner with annular feed ducts c_4 and c_5 arranged concentrically around the reaction gas pipe b , for feeding the gases for the auxiliary reaction, said annular ducts being provided with partition walls h for imparting a spinning motion.

Fig. 6 shows a combination of the devices of Figs. 4 and 5, i.e. a feeding device in which the twist-imparting helical partition walls g or h are provided both in the annular feed pipes c_4 and c_5 and in the central feed pipe b for the reaction gases. The guide blades in these examples are disposed in such a way that the spin of one of the gases of the auxiliary reaction produced in the intermediate feed pipe c_4 is opposed to both that produced by the partition walls g of the reaction gas feed pipe b and to the spin of the other gas of the auxiliary reaction produced by the partition surfaces h of the outer feed pipe c_5 . By varying the positions of the guide blades in the various feed pipes, twisting motions can be produced which are directed equally or oppositely in any desired combination.

Figs. 7 and 8 show in a front view two burners in which several non-concentric feed ducts c_6 and c_7 for at least one of the gases of the auxiliary reaction are arranged around the central feed pipe b of the reaction gas. The pipe axes in this case are directed so skew against the axis of the central reaction gas mixture feed pipe b that they do not intersect it. In Fig. 7, this arrangement is adopted only for the feed pipes c_6 of one of the gases of the auxiliary reaction while an annular feed pipe c_4 is provided for the other gas. In Fig. 8, on the other hand, the other gas is also fed to the reaction chamber by feed pipes c_7 arranged skew in the same manner as at c_6 . In these arrangements it is also

possible to impart a spin to the reaction gas mixture by providing its feed pipe with oblique partition walls g as in Fig. 4. The heating elements possibly provided for heating the walls of the reaction chamber are shown by way of example in the form of electrical heaters i in Fig. 2.

Fig. 9 is a sectional view of a burner with concentric feed pipes b , c_4 and c_5 for the reaction gas mixture and the gases for the auxiliary reaction, respectively, in which the outermost gas stream can be directed into the reaction gas stream. To this end the outermost feed pipe c_5 is provided with a conically shaped end member k . Fig. 10 shows a sectional view of a burner which is similar to Fig. 9, except that the two feed pipes for the gases for the auxiliary reaction c_4 and c_5 are provided with a conically shaped end member k .

In the embodiment of Fig. 9 the walls of the conically shaped ends of the outermost feed pipe are straight conical surfaces, while in Fig. 10 the walls of the conically shaped ends of the feed pipes are curved parabolically. Instead of a parabolical curvature a circular, elliptical or other curvature would also be possible.

Burners such as those shown in Figs. 9 and 10 may also be provided with twist-imparting means such as those shown in Figs. 4 to 6.

The process according to the invention and its materialisation in the aforescribed devices will now be explained in greater detail by way of some examples.

EXAMPLE 1.

Through a feed pipe b a mixture of 1 part by weight of titanium tetrachloride vapor and 3 parts by volume of air enriched to an oxygen content of 50 percent oxygen was introduced into a reaction chamber similar to that shown in Fig. 2, with a temperature of 100°C and a velocity of 10m/sec. In a later arranged burner, consisting of the concentric feed pipes c_2 and c_3 , 0.1 part by volume of benzine vapor was burnt with 1.5 part by volume of oxygen, adjusting the ignition flame in such a way that its tip came into intimate contact with the reaction gas current. A yield of 96 percent of titanium oxide with an average particle size of 1 micron was obtained.

EXAMPLE 2.

Through the central pipe b of a burner device as per Fig. 3 a reaction gas consisting of 1 part by volume of titanium chloride vapour and 0.8 parts by volume of oxygen was introduced into a reaction chamber as per Fig. 3 with an outlet velocity of 5 m/sec. and a temperature of 150°C. 1 part by volume of carbon monoxide was introduced through the annular feed duct c_4 and 1 part by volume of oxygen was introduced through the feed pipe c_5 and ignited. The outlet velocity of the latter two gases amounted to 8 m/sec. The reaction gas mixture was ignited at a distance

of 3 cms. from the outlet opening. A yield of 99 percent of a product with an average particle size of 0.75 micron was obtained.

EXAMPLE 3.

5 Through a burner as per Fig. 6, the following gases were introduced into a reaction chamber as per Fig. 3: Through the central tube b a mixture of 1 part by volume of titanium chloride vapor and 1.5 part by
10 volume of oxygen (temperature 250°C, velocity 20 m/sec.); through the feed duct c₁ 0.8 part by volume of carbon monoxide, and through the feed duct c₂ 0.4 parts by volume of oxygen. The reaction gas ignited at a distance
15 of 5 to 10 cms. A yield of 99 percent of a product of 0.5 micron particle size was obtained.

EXAMPLE 4.

20 Through the same device as in the preceding example there were introduced: Through the central pipe a mixture of 1 part by volume of zirconium tetrachloride vapor and 1.5 part by volume of oxygen (with 300°C and a velocity of 20 m/sec.). With conditions which for the rest were the same as in Example 3, a
25 zirconium oxide of 0.75 microns average particle size was obtained.

EXAMPLE 5.

30 Through a burner as per Fig. 8 there was introduced into a reaction chamber as per Fig. 3: In the central feed pipe b a mixture of 1 part by volume of aluminium chloride vapour and 3 parts by volume of oxygen (discharge temperature 250°C, velocity 20 m/sec.),
35 through the feed ducts c₁ 1 part by volume of town or lighting gas and through the feed ducts c₂ 2 parts by volume of oxygen. The aluminium oxide produced with a yield of 96 percent had an average particle size of 1
40 micron.

EXAMPLE 6.

The following gases were fed through a device, i.e., a burner, as per Fig. 9 whose cone angle (2α) is 60°, into a reaction chamber of
45 the type shown in Fig. 3: Through the central pipe b a mixture of 1 part by volume of titanium chloride vapor and 1.3 parts by volume of oxygen with a temperature of 120°C and an exit velocity of 20 m/sec., through
50 the feed duct c₁ 1 part by volume of carbon monoxide with a velocity of 4 m/sec., and through the feed duct c₂ 0.5 parts by volume of oxygen with an exit velocity of 5 m/sec.. The reaction gas ignited at a distance of 1 cm.
55 A titanium oxide of 0.4 microns average size of the single particle was obtained.

EXAMPLE 7.

There was delivered into the same reaction chamber as per example 6, through a burner
60 as per Fig. 10, with a cone angle 2α of the intermediate feed duct of 120° and of the outer feed duct of 90°. In the central pipe b a mixture of 1 part by volume of titanium chloride vapour and 1 part by volume of oxy-
65 gen (temperature 150°C, exit velocity 15

m/sec.), through the feed pipe c₁ 1 part by volume of carbon monoxide (velocity 3 m/sec.) and through the feed duct c₂ 0.8 parts by volume of oxygen with 4 m/sec. A product with an average size of the single crystal
70 of 0.2 microns was obtained.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—
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1. A process of producing finely divided oxide of titanium, silicon, zirconium or aluminium by the decomposition by burning at high temperatures, of the corresponding volatile chloride in the presence of oxygen-containing gases, comprising the steps of supplying into a reaction chamber a reaction gas mixture of the chloride in the vapour state and oxygen-containing gas at a temperature above the dewpoint of the chloride in the reaction gas mixture but not exceeding 500°C.,
85 a combustible gas introduced separately from the reaction gas mixture, and oxygen-containing gas additional to the oxygen-containing gas in the reaction gas mixture and introduced separately from the reaction gas mixture, the additional oxygen-containing gas introduced being at least sufficient to ensure that the combustible gas would burn under flaming conditions in the absence of the reaction gas mixture and being at least sufficient (together with the oxygen-containing gas in the reaction gas mixture) to ensure complete combustion of the chloride, the gases being so introduced into the reaction chamber and in such proportions and amounts that the combustible gas and the additional oxygen-containing gas (introduced separately from the reaction gas mixture) are at least in part admixed before such admixture meets the reaction gas mixture and that the said combustible gas and the additional oxygen-containing gas serve to maintain the burning of the reaction gas mixture in a flaming state.
90

2. A process as claimed in claim 1, in which the composition of the reaction gas mixture, its temperature and exit velocity are chosen in such a way that the flame which is formed is kept at a minimum distance of 1 mm. up to a maximum distance of 1 m. from the outlet opening of the reaction gases in the reaction chamber.
100

3. A process as claimed in claim 1 or 2, in which at least part of the additional oxygen-containing gas is introduced separately from the combustible gas.
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4. A process as claimed in any one of the preceding claims, in which the combustible gas is carbon monoxide.
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5. A process as claimed in any one of claims 1 to 3, in which the combustible gas is a hydrogen-containing gas.
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6. A process as claimed in any one of claims 1 to 3, in which the combustible gas is a hydrogen-containing gas.
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130

7. A process as claimed in any one of the preceding claims, in which part of the oxygen required for combustion of the combustible gas is fed in admixture with the chloride vapour. 5
8. A process as claimed in any one of the preceding claims, in which the combustible gas and the additional oxygen-containing gas are supplied in the form of one or more streams surrounding the reaction gas mixture. 10
9. A process as claimed in claim 8, in which either the combustible gas or the additional oxygen-containing gas is directed to intersect with the direction of the reaction gas mixture stream. 15
10. A process as claimed in claim 9, in which both the combustible gas and the additional oxygen-containing gas are directed to intersect with the direction of the reaction gas mixture stream. 20
11. A process as claimed in any one of claims 3 to 10, in which means are provided for twirling or spinning the combustible gas and additional oxygen-containing gas during combustion of the said gases. 25
12. A process as claimed in any one of the preceding claims, in which means are provided for twirling or spinning the reaction gas mixture being fed into the reaction chamber.
13. A process for the production of finely divided oxide of titanium, silicon, zirconium or aluminium substantially as herein described and with reference to any one of the examples 1 to 7. 30
14. A process for the production of finely divided oxide of titanium, silicon, zirconium or aluminium using an apparatus constructed and arranged substantially as herein described and shown in any one of Figures 1 to 10 of the accompanying drawings. 35
15. Finely divided oxide of titanium whenever prepared or produced by the process claimed in any one of claims 1 to 14. 40
16. Finely divided oxide of zirconium whenever prepared or produced by the process claimed in any one of claims 1 to 14. 45
17. Finely divided oxide of silicon whenever prepared or produced by the process claimed in any one of claims 1 to 14.
18. Finely divided oxide of aluminium whenever prepared or produced by the process claimed in any one of claims 1 to 14. 50

Dated this 10th day of February, 1949.

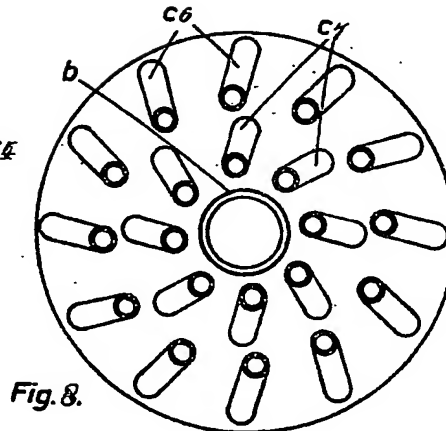
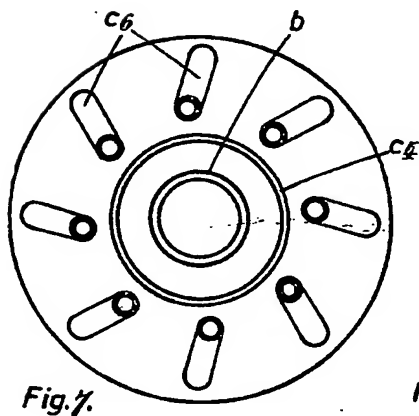
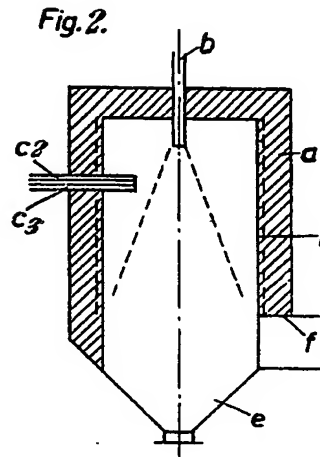
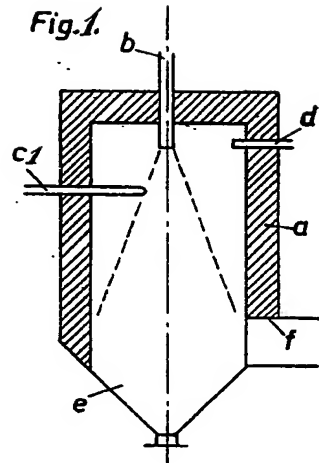
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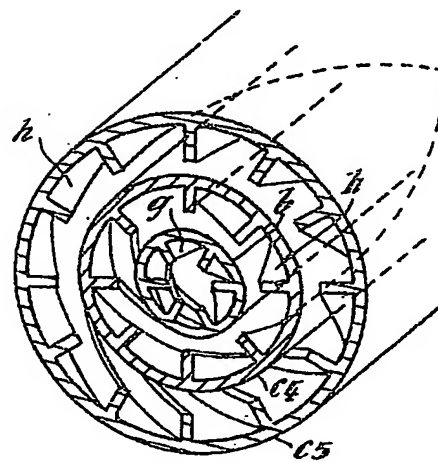
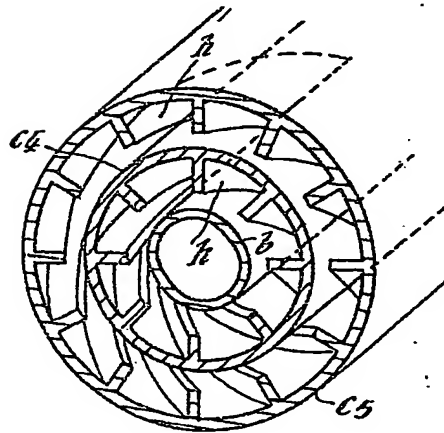
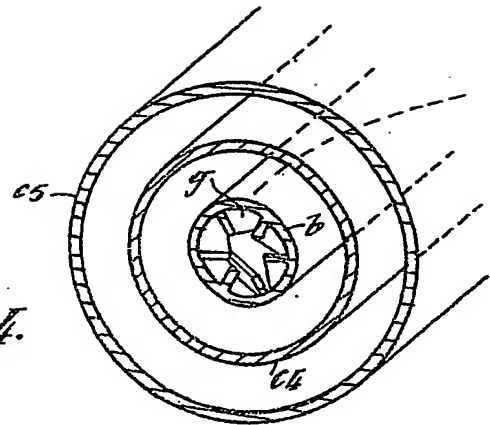
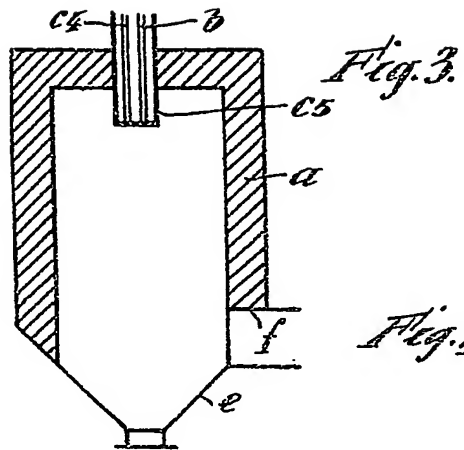
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SHEETS 1 & 3





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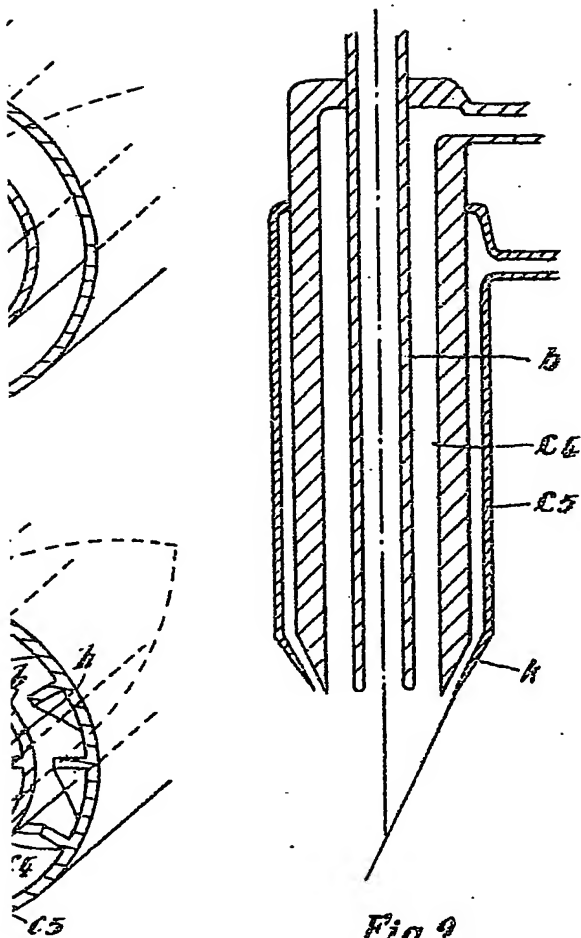


Fig. 9

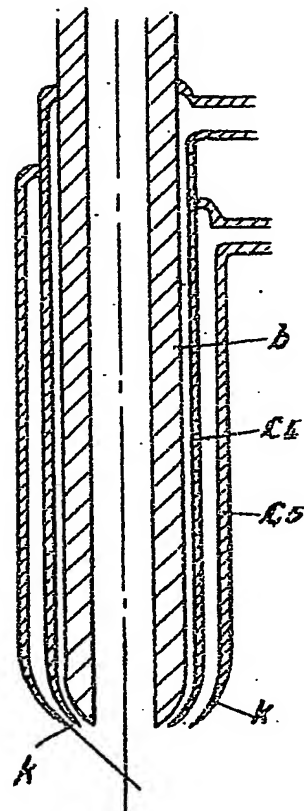


Fig. 10.

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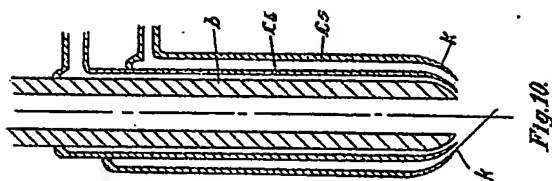


Fig. 10.

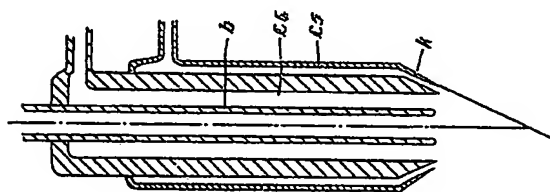


Fig. 9.

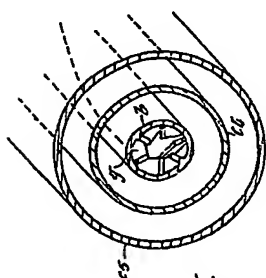


Fig. 8.

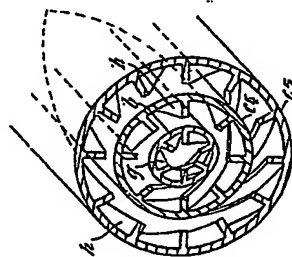


Fig. 6.

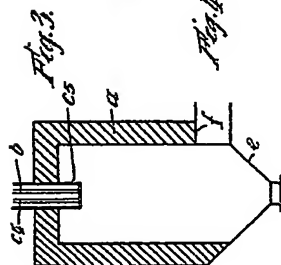


Fig. 3.

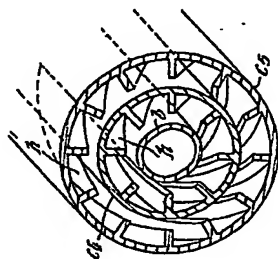


Fig. 5.

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